

Republic of Kenya

EDICT OF GOVERNMENT

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KS 87 (2012) (English): Specification for
hydrochloric acid (Draft Standard)



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Specification for hydrochloric acid

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Specification for hydrochloric acid

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P R E F A C E

This Kenya Standard was prepared by the Technical Committee on Industrial solvents and chemicals under the authority of Kenya Bureau of Standards.

The standard applies to technical and analytical grades of hydrochloric acid.

During the preparation of this standard, reference was made to the following publications and acknowledgement is made for their assistance with thanks:

BS 3993: 1966 – Specification for hydrochloric acid commercial.

IS 265:1976 – Specification for hydrochloric acid.

ISO/R 904:1976 – Hydrochloric acid for industrial use – Determination of total acidity – Volumetric method.

PUBLIC REVIEW DRAFT FEBRUARY 2012

KENYA STANDARD
SPECIFICATION FOR HYDROCHLORIC ACID

1. Scope

- 1.1 This Kenya Standard prescribes the requirements, sampling and methods of test for hydrochloric acid for industrial use.

2. Grades

- 2.1 There shall be two grades of hydrochloric acid, namely technical and analytical reagent grades.

3. Requirements

3.1 Description

- 3.1.1 Technical grade – The material shall be a clear, colourless or light yellow liquid, free from visible impurities.

- 3.2 The material shall also comply with the requirements given in Table 1. Reference to test methods in Appendix A is given in Column 6 of Table 1.

Table 1 – Requirements for hydrochloric acid

Sl. No.	Characteristic	Requirement		Method of test Ref. to Cl. No. in Appendix A
		Technical (Tech) Grade	Analytical reagent (AR) Grade	
(1)	(2)	(3)	(4)	(5)
(i)	Total acidity as HCl, per cent by mass, min	32	35.4	A2
(ii)	Residue on ignition, per cent by mass, max	0.1	0.0005	A3
(iii)	Sulphates as H ₂ SO ₄ , per cent by mass, max.	0.02	0.0002	A4
(iv)	Iron as Fe, per cent by mass, max.	0.005	0.00005	A5
(v)	Free chlorine and bromine as Cl, per cent by mass, max.	0.02	0.0002	A6
(vi)	Sulphates as SO ₂ , per cent by mass, max.	0.05	0.0001	A7
(vii)	Heavy metals as Pb, per cent by mass, max.	0.0005	0.0001	A8
(viii)	Arsenic as As, per cent by mass, max.	0.0002	0.000002	A9
(ix)	Ammonium compounds as NH ₄ percent by mass max		0.0003	A10
(x)	Mercury as Hg, per cent by mass, max.	0.005	0.000002	A11

4. Packaging and marking

4.1 Packaging

4.1.1 The material shall be packed in clean containers that do not react with it.

4.1.1.1 When hydrochloric acid is supplied in screw-stoppered stone bottles, glass carboys, polyethylene carboys or hard-rubber drums, the containers shall be fitted with leak-tight stoppers or screw caps and where necessary provided with rubber washers.

4.1.1.2 Hydrochloric acid of analytical reagent grade shall be supplied in glass bottles fitted with leak-tight stoppers or screwed caps and no washers shall be used in closing the bottles. The use of suitable caps over the stoppers is recommended. It may also be packed in plastic containers.

4.2 Marking

4.2.1 The following information shall be suitably marked in red letters, not less than 2.5 mm high, on the packages:

- (i) The name of the acid
- (ii) The manufacturer's name/trade mark and address
- (iii) The grade and net content

- (iv) Country of origin
- (v) Batch number
- (vi) Date of manufacture

The packages shall also prominently display the words 'CORROSIVE HANDLE WITH CARE!'.

- 4.2.2 In case of analytical reagent grade, the actual analysis of the material with respect to the characteristics laid down in Column 4, Table 1 shall also appear on the label.

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Quality of reagents

Unless specified otherwise, pure chemicals and distilled water complying with KS 03-88 shall be used in the tests.

NOTE – 'Pure chemicals' shall mean chemicals that do not have impurities which affect the results of analysis.

ANNEX A Determination of total acidity

A1.1 Apparatus

A1.1.1 Pipette – (see Figure 1). If this pipette is not available, a weighing bottle fitted with ground glass stopper or a glass ampoule (see Figure 2) may be used.

A.1.1.2 Filter Paper

A1.2 Reagents

A1.2.1 Standard Sodium hydroxide solution – 1N.

A1.2.2 Bromocresol green indicator solution – Dissolve 0.1 g of bromocresol green in 100 ml of 95 per cent (v/v) ethanol.

A1.2.3 Phenolphthalein indicator solution – Dissolve 0.1 g of phenolphthalein in 100 ml of 60 per cent ethanol.

A1.3 Procedure

A1.3.1 If an ordinary weighing bottle is used for weighing the sample, then accurately weigh about 2 g to 3 g of the material in it.

If a glass ampoule is used, take sufficient amount of sample in a beaker or flask. Slightly heat on a flame the bulb of the glass ampoule previously weighed to the nearest 0.1 mg. Immerse the capillary end of the ampoule into the beaker or flask containing the test sample and ensure that the bulb is filled up to about two thirds of its volume during cooling (2 ml to 3 ml approximately).

Withdraw the ampoule and carefully wipe the capillary end with filter paper. Seal the capillary end in an oxidizing flame, without loss of glass. Remove the glass ampoule from the flame and allow to cool. Wash the capillary and wipe carefully with filter paper. Weigh the ampoule to the nearest 0.1 mg and calculate by difference the mass of the test portion.

A1.3.2 If the sample was weighed in the pipette or weighing bottle, transfer the sample to a conical flask and dilute to 100 ml with water. If the sample was weighed in an ampoule, place it into the conical flask containing 200 ml of cold water. Stopper the flask while cooling, shake to break the ampoule containing the test portion. Keep cooling and shaking until the vapours are completely absorbed. Remove the stopper and rinse it with water, collecting the washings in the conical flask. By means of a glass rod, mince the fragments of the ampoule and in particular the capillary which may have remained intact inspite of shaking. Withdraw the glass rod and wash with water, collecting the washings in the conical flask.

A1.3.3 Add two drops of bromocresol green or phenolphthalein indicator to the solution in the beaker or flask and titrate to the end point with the standard sodium hydroxide solution.

A1.4 Calculation – Total acidity (as HCL), per cent by mass

$$= \frac{v \times N \times 3.646}{m}$$

where,

v = volume in ml of standard sodium hydroxide solution used for titration;

N = normality of standard sodium hydroxide solution; and

m = mass in g of the sample taken for the test.

ANNEX B

Determination of residue on ignition

B.1 Reagent

B.1.1 Concentrated sulphuric acid

B.2 Procedure – In a platinum dish previously ignited at $800 \pm 25^{\circ}$ C, cooled in a desiccator and weighed, weigh to the nearest 10 mg approximately 100 g of the test sample. Evaporate most of the acid (the final volume should amount to about 5 ml to 10 ml) by carefully heating the dish containing the test portion (on a sand-bath, for example). Allow to cool to room temperature, add 1 ml of concentrated sulphuric acid and heat to dryness. Place the dish containing the residue in an electric furnace heated at the dish from the furnace, cool in a desiccator and weigh. Repeat the heating, cooling and weighing to constant mass.

B.3 Calculation – Residue on ignition, per cent by mass.

$$= \frac{m_1 \times 100}{m_2}$$

where,

m_1 = mass in g of the residue weighed; and

m_2 = mass in g of the sample taken for the test.

ANNEX C

Determination of sulphates

C.1 For technical grade

C.1.1 Sodium carbonate – Analytical reagent grade.

C.1.2 Barium chloride solution – Approximately 10 per cent (m/v).

C.1.3 Filter paper

C.1.4 Procedure – Weigh to the nearest 0.01 g about 10 g of the material (m_1) in a porcelain dish, add 0.5 g of sodium carbonate and evaporate to dryness. Moisten the residue with about 1000 g of the material accurately weighed (m_2), add 200 ml of boiling water and filter if necessary. Bring the contents to boil over a low flame and add slowly, with stirring, 5 ml of hot barium chloride solution. Boil the contents for 2 min and allow the precipitate to settle for 4 h. Filter the supernatant liquid through a tared sintered glass crucible (G No. 4) or a tared Gooch crucible and transfer the precipitate carefully into the crucible. Wash thoroughly with hot water till the washings are free from chlorides. Heat the crucible at 110°C to constant mass.

C.1.5 Calculation – Sulphates (as H_2SO_4), per cent by mass

$$= \frac{42.02m}{m_1 + m_2}$$

where,

m = mass in g of the precipitate;

m_1 = mass in g of the sample taken initially; and

m_2 = mass in g of the sample added subsequently

C.2 For analytical reagent grade

C.2.1 Reagents

C.2.1.1 Sodium carbonate – Analytical reagent grade.

C.2.1.2 Hydrochloric acid – Approximately 1N, sulphate-free.

C.2.1.3 Barium chloride solution – Approximately 10 per cent (m/v)

C.2.1.4 Standard sulphate solution – Dissolve 0.178 g of potassium sulphate in water and make up the volume to 1000 ml. Dilute 100 ml of the solution in a graduated flask to 1000 ml. One milliliter of the diluted solution contains 0.01 mg of sulphate (as H_2SO_4).

C.3 Procedure

C.3.1 To 20.00 g of the material add 20 mg of sodium carbonate and evaporate to dryness. Take up the residue with 5 ml of water and 0.5 ml of hydrochloric acid. Filter, wash with water to make up to 10 ml and add 1 ml of barium chloride solution. Carry out a control

test in the same manner using 4 ml of standard sulphate solution in place of the material. Stir the two solutions and compare the turbidity produced.

- C.3.2 The limit prescribed in Table 1 shall be taken as not having been exceeded if the turbidity produced with the material is not greater than that produced in the control test.

ANNEX D

Determination of iron

D.1 Method A (Bipyridyl method)

D.1.1 Apparatus

- D.1.1.1 Photometer – Any spectrophotometer suitable for measurement at a wavelength of about 522 nm, or a photoelectric absorptiometer.

D.1.2 Reagents

- D.1.2.1 Dilute hydrochloric acid – 1N approximately.

- D.1.2.2 Hydroxylammonium chloride solution – Dissolve 10 g of hydroxylammonium chloride in water and dilute to 100 ml.

- D.1.2.3 2, 2'-bipyridyl solution – Dissolve 1 g of 2,2'-bipyridyl in 10 ml of dilute hydrochloric acid (1N) and dilute to 100 ml with water.

- D.1.2.4 Standard iron solution A – Dissolve 0.7022 g of ferrous ammonium sulphate $[\text{FeSO}_4(\text{NH}_4)\text{SO}_4 \cdot 6\text{H}_2\text{O}]$ in water in a 1000 ml.

Volumetric flask, add 4 ml of concentrated sulphuric acid and make up to the mark with water. One milliliter of this solution contains 0.1 mg of iron (as Fe).

- D.1.2.5 Standard iron solution B – Take 100 ml of the standard iron solution A (see Clause D.1.2.4) and dilute to 100 ml with water in a 1000 ml volumetric flask. This dilute solution should be prepared fresh. One milliliter of this solution contains 0.01 mg of iron (as Fe).

- D.1.2.6 Ammonium acetate solution – Dissolve 30 g of ammonium acetate in water and dilute to 100 ml.

D.1.3 Procedure

- D.1.3.1 Weigh accurately about 50 g of the sample in a platinum or silica dish (100 ml capacity), place on a boiling water bath and carefully evaporate to dryness. Cool, take up with 2 ml of hydrochloric acid and 25 ml of water, and heat to facilitate dissolution. Transfer quantitatively to a 100 ml one-mark volumetric flask, dilute to the mark, mix and filter if necessary. Transfer an aliquot to the sample solution containing between 50 µg and 500 µg of iron to a 100 ml one-mark volumetric flask.

Dilute to approximately 50 ml if necessary and then add successively 2 ml of hydrochloric acid solution, 2 ml hydroxylammonium chloride solution and after 5 min, 5 ml of ammonium acetate solution and 1 ml of 2,2'-bipyridyl solution. Dilute to the mark, mix and wait for 10 min. Carry out the measurement on the spectrophotometer at a wavelength of about 522 nm, adjusting the instrument to zero optical density using as reference the blank test.

D.1.3.2 Blank test – At the same time as the analysis, carry out a blank test using the same procedure and quantities of all reagents employed in the test.

D.1.3.3 Prepare a calibration curve by taking the quantities of standard iron solution indicated below in series of eleven 100 ml volumetric flasks:

Volume of standard Iron Solution B	Corresponding mass Iron (Fe)
ml	µg
0*	0
5.0	50
10.0	100
15.0	150
20.0	200
25.0	250
30.0	300
35.0	350
40.0	400
45.0	450
50.0	500

Add to each volumetric flask an amount of water sufficient to dilute to approximately 50 ml, then 2 ml of dilute hydrochloric acid solution, 2 ml of hydroxylammonium chloride solution and after 5 min, 5 ml of ammonium acetate solution and 1 ml of 2,2'-bipyridyl solution. Dilute to the mark, mix thoroughly and wait for 10 min.

D.1.3.4 Carry out the photometric measurements as in Clause D.1.3.1 adjusting the instrument to zero optical density using as reference the compensation solution. Prepare a calibration curve having, for example the iron content in micrograms per 100 ml of the standards matching solution as abscissa and the corresponding values of absorbance as ordinate.

D.1.4 Calculation – Iron content (as Fe), per cent by mass

$$= \frac{m_1 \times 100 \times 100}{v \times m}$$

where,

m_1 = mass in g of iron determined in the aliquot of sample solution;

v = volume in ml of the sample taken for colour reaction;

m = mass in g of the test portion

* Compensation solution

D.2 Method B (Thiocyanate Method)

D.2.1 Apparatus

D.2.1.1 Nessler cylinders – 50 ml capacity.

D.2.2 Reagents

D.2.2.1 Ammonium persulphate

D.2.2.2 Butanolic potassium thiocyanate solution – Dissolve 10 g of potassium thiocyanate in 10 ml of water. Add sufficient n-butanol to make up to 100 ml and shake vigorously till the solution is clear.

D.2.2.3 Standard iron solution B – Same as in Clause D1.2.5.

D.2.3 Procedure

D.2.3.1 For technical grade – Dilute 1000 g of the material to 100 ml with water. Transfer 10 ml of this solution to a Nessler cylinder, add about 30 mg of ammonium persulphate and 15 ml of butanolic potassium thiocyanate solution. Make up to 50 ml, shake vigorously for about 30 s and allow the layers to separate. Carry out a control test in another Nessler cylinder using the same reagents and maintaining the same total volume, with 2 ml of the standard iron solution B in place of the material. Compare the intensity of the colour produced in the butanol layers in two cylinders.

D.2.3.2 For Analytical Reagent grade – Weigh 50.0 g of the material and evaporate it almost to dryness. Dilute it to 30 ml, add about 30 mg of ammonium persulphate and 15 ml of butanolic potassium thiocyanate solution. Make up to 50 ml, shake vigorously for test in another Nessler cylinder using 2 ml of standard iron solution B. Compare the intensity of the colour produced in the butanol layers in the two cylinders.

D.2.4 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced with the material is not greater than that produced in the control test.

D.3 AOAC/AAS as an alternative method

ANNEX E

Determination of free chlorine and bromine.

E.1 For technical grade

E.1.1 Reagents

E.1.1.1 Potassium iodide – Crystals.

A6.1.1.2 Starch solution – Make a paste of 0.5 g of starch with 2.5 ml of water. Pour the paste in small quantities into 200 ml of water. Boil for 15 min with stirring. Preserve in small containers previously sterilized in boiling water for 2 h.

E.1.1.3 Potassium iodide solution – 10 per cent.

E.1.1.4 Standard sodium thiosulphate solution – 0.1 N.

E.1.2 Procedure

- E1.2.1 Preliminary test – Place about 20 ml of the test sample in a 100 ml conical flask, add 50 ml of water, one crystal of potassium iodide, 0.5 ml of starch solution and stir. If a blue colour indicating iodine liberation appears follow the procedure prescribe in Clause E.1.1.2 for determination of free chlorine and bromine. If no colour appears, proceed to determination of sulphates as in Annex C.

ANNEX F Determination of heavy metals as Lead

- F.1 Use AAS method

ANNEX G Method for the determination of arsenic content

- G.1 Use AAS method

ANNEX H Determination of ammonium compounds

H.1 Reagents

- H.1.1 Sodium hydroxide solution – 5N, ammonia-free.

- H.1.2 Standard ammonium solution – Dissolve 2.97 g of ammonium chloride in water and dilute to 1000ml. 1ml of this solution contains 1 mg of ammonia. Dilute 1 ml of this solution to 100 ml with water immediately before use to get 0.01 mg of ammonia per ml.

- H.1.3 Nessler's reagent – Dissolve 35 g of potassium iodide and 12.5 g of mercuric chloride in 800 ml of water and add a saturated solution of mercuric chloride until a slight permanent precipitate is produced. Then add 120 g of sodium hydroxide solution and sufficient water up to 1000 ml. Shake the solution occasionally for several days. Allow to settle and decant the clear liquid.

- H.2 Procedure – Dilute 2.8 ml (3.3 g) of the material with water to about 20 ml and add sodium hydroxide solution, until alkaline to litmus paper (about 7 ml is required). Dilute to 50 ml with water. Prepare standard by diluting 1 ml of standard ammonium solution to 50 ml with water. To both solutions add 2 ml of Nessler's reagent and compare after 1 min.

- H.3 The limit prescribed in Table 1 shall be taken as not having been exceeded if the yellow colour produced, if any, in the test with the material is not deeper than that of the standard.

Alternatively, use High Performance Liquid Chromatography (HPLC) method.

ANNEX I
Determination of mercury

I.1 Use AAS method.

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